

Saimaa University of Applied Sciences
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Degree Programme in Paper Technology

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DETERMINATION OF STRENGTH PROPERTIES OF NORWAY SPRUCE AFTER SULPHATE COOKING AND OXYGEN DELIGNIFICATION

Bachelor's Thesis 2011

ABSTRACT

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Determination of Strength Properties of Norway Spruce After Sulphate cooking Process and Oxygen Delignification, 55 pages

Saimaa University of Applied Sciences, Imatra

Unit of Technology, Degree Programme in Paper Technology

Bachelor's Thesis, 2011

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The aim of this thesis was to analyze the strength properties of Norway spruce kraft pulp required for the paper making process. In the theoretical part, the properties of Norway spruce were introduced, and its comparison properties with pine and birch were physical and chemical aspects of different wood species and their properties by Saimaa University of Applied Sciences, Imatra.

In the experimental part, there are two different types of kraft pulp were studied; one pulp with sulphate cooking process treatment, and another one with the oxygen delignification. All these pulp were produced from the fresh Norway spruce chips in liquid circulated batch digester by kraft pulping process. Screened pulps were beaten by PFI mill in 0, 2000, 4000, 6000 and 8000 revolution degrees. Fiber length was measured by Kajaani FS 300; the water retention test was also done with the specific equipment. Paper sheets were made by rapid Köthen sheet mould. Tear strength, burst strength, tensile strength, thickness and weight were measured from the paper sheets.

Keywords: Sulphate Cooking, Oxygen Delignification, Norway Spruce, Beat, Kraft Pulp, Strength.

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1 INTRODUCTION

In the final thesis, the strength properties of paper and its comparison with sulphate cooking process and oxygen delignification treatment are the main purpose of this thesis. The pulps were beaten to different revolution degree; comparing how the different beating degree affects the paper's properties.

In the theoretical part, the properties of Norway spruce were introduced, the structure of wood, chemical and physical properties of Norway spruce were explained, general comparison properties of birch, pine and Norway spruce, the different fibers dimensions were introduced too. In the chemical pulp part, the sulphate cooking process and oxygen delignification treatment were done for the Norway spruce chemical pulps, beating and its effect on properties of paper are also briefly introduced in this part.

In the experimental part, kappa analysis, white liquor analysis, water retention and fiber length test and so on were done in the laboratory of Saimaa University of Applied Sciences.

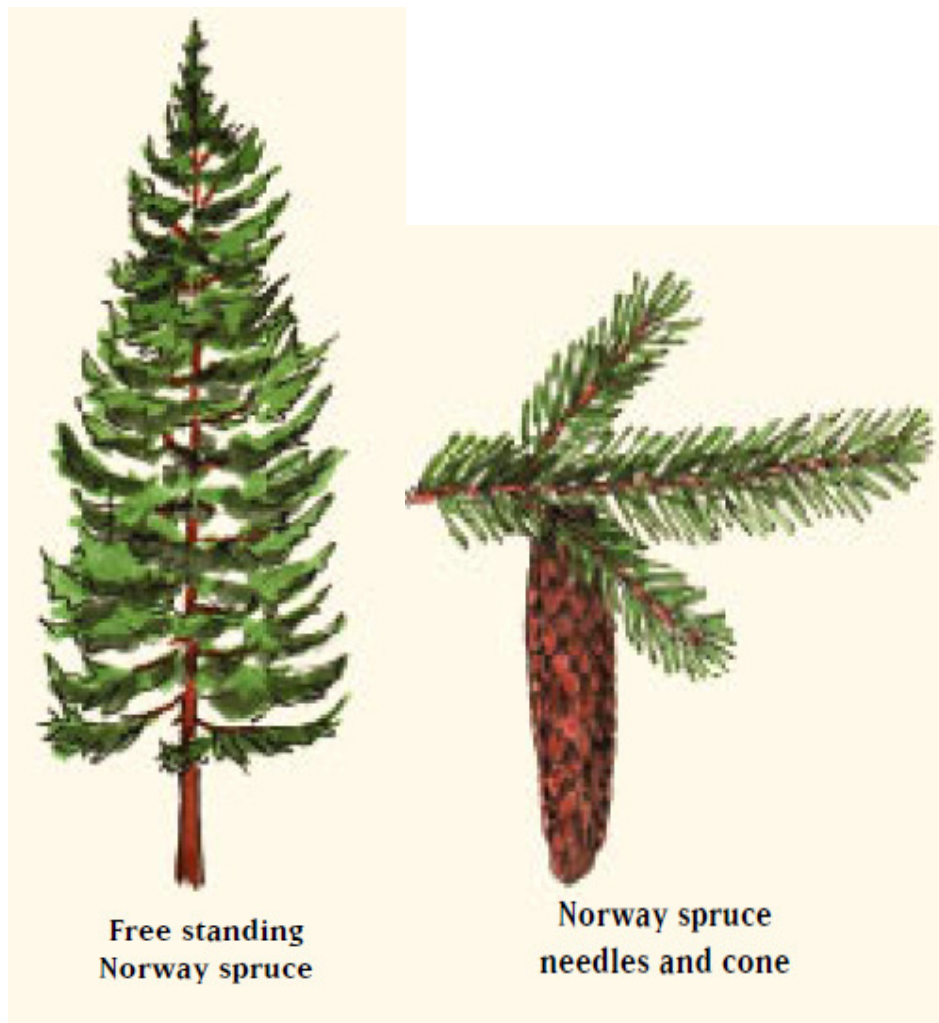
2 SPRUCES

2.1 General

There are more than 100 forms and varieties of spruce; Norway spruce is one of the most important species in Europe, although not native to the western hemisphere, the Norway spruces are commonly planted in Canada and north United States. Norway spruce is a part of the pine family. Nowadays the Norway spruce is widely used for the Christmas tree. It has a very strong central leader with horizontal side branches that have a sweeping pendulous character as the tree matures. Generally, the diameter of spruce can reach as much as two feet on older trees. It has dark green crown with a triangular shape, and sharp blunt at the tip. At the base of each needle is a twig-like projection (sterigmata) which remains after the needle is lost. (Karst,2006). Normally, spruces have rectangular rather than flat needles, and cones which hang down rather than stand erect on the stem. Additionally, spruce cones fall from the tree after seeds are disseminated, whereas fir cones disintegrate. There are many small-diameter roots originate from the base of the trunk and they are often found fairly close to the surface of the soil. It is shallow of the root system, specially close to the trunk which makes growing grass difficult. (Ohio Trees)

In its native range, Norway spruce occurs in pure stands; transitional stands mixed with Scotch pine or mixed stands with European beech. Scattered Norway spruce occurs in seral stands of European aspen (*Populus tremula*) or hairy birch (*Betula pubescens*). Classification systems for Scandinavian forests where Norway spruce and/or Scotch pine are the major species are based on ground vegetation. Good sites for Norway spruce occur on *Oxalis-Myrtillus* types and fair sites are indicated by *Myrtillus*. *Vaccinium* types are usually rather barren and not suited for good spruce growth. Understory

species most often associated with Norway spruce in Poland include raspberry (*Rubus idaeus*) and European mountain-ash (*Sorbus aucuparia*). Mature Norway spruce forests typically have very little ground layer vegetation. (Norway spruce - *Picea abies*), A picture of Norway spruce is shown in: picture 1. (Ohio Trees.)



Picture 1: the shape of Norway spruce (USDA, ARS, National Genetic Resources Program.)

Norway spruce wood is strong, soft, straight- and fine-grained, and easily worked. It is not durable in contact with soil. It is widely used for construction, pulp, furniture, and musical instruments. Norway spruce is one of the most

common and economically important coniferous species in Europe and Scandinavia. In Maine, thinned material and standing dead Norway spruce produced pulp of good strength as reported in a study of the pulp potential of seven softwoods. (Marek FABRIKA, 2006, page 127-136)

2.2 Structure and Applications of Norway Spruce

The outer layer of the secondary wall is c. 0.12 μm thick in early wood and 0.38 μm thick in latewood cell walls of Norway spruce. tracheids and represents approximately 10% of the total cell wall thickness. The outer layer is much thicker in compression wood tracheids than in normal wood (Timell 1986). Since the outer layer is rather thin, considerable difficulties arise when trying to determine its architectural organization. Therefore, outer layer ultra structure has not been investigated in as much detail as the thick middle layer of the secondary wall which represents about 80% of the total cell wall thickness (Fengel & Stoll 1973). However, the outer layer has a very important role for pulp fibers since it will be in direct contact with chemicals or enzymes and during beating it will be subjected to large mechanical forces. The thickness and micro fibril angle of the outer layer has been shown to be important for the transverse elastic modulus of Norway spruce tracheas (Global Ecology and Biogeography 1999.)

The application of Norway spruce is used widely in the world. The wood is strong for its weight, odorless, but resinous. It is a very important raw material in the paper and pulp manufacture. Resinous bark exudations furnish what is known as "Burgundy pitch" which is the basic material for a number of varnishes and medicinal materials. New leafy shoots can be used for brewing spruce beer. The wood is also used in violin sound boards. (IAWA Journal)

2.3 Chemical Composition of Norway Spruce

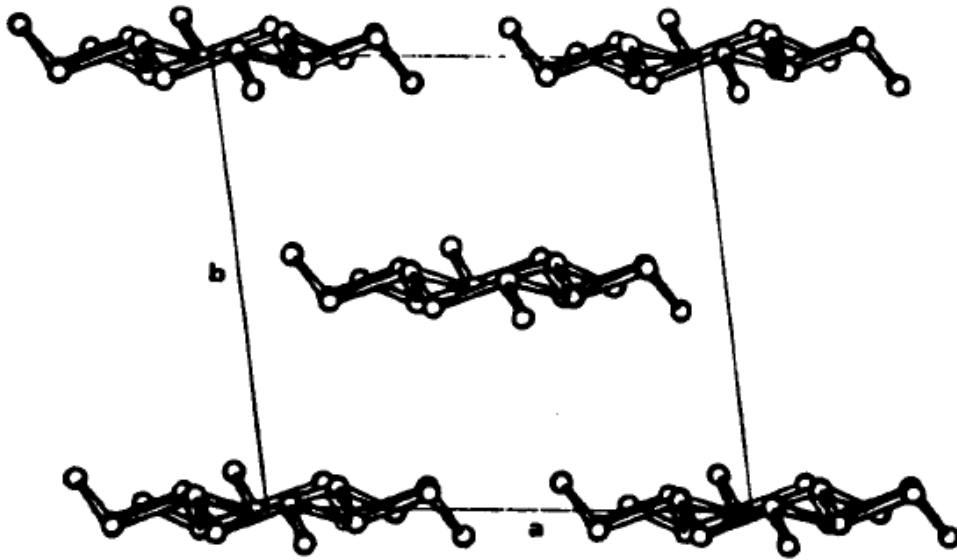
The chemical compositions of Norway spruce are mainly containing three compositions; they are cellulose, hemicelluloses and lignin. In fact, cellulose and hemicelluloses are called carbohydrate; it means there are two major chemical compositions in wood. Carbohydrate (65%-75%) and lignin (18%-35%) are complex and polymeric materials. You can not give a exactly definition of the chemical composition of wood, the reason is that chemical composition is different with the tree part(root, stem, or branch), geographic location, climate and so on. Extractives are also containing in the wood, such as resin, in the chemical pulping process, the extractives are removed. (Forest Products Laboratory, Madison, WI 53705)

2.3.1 Cellulose

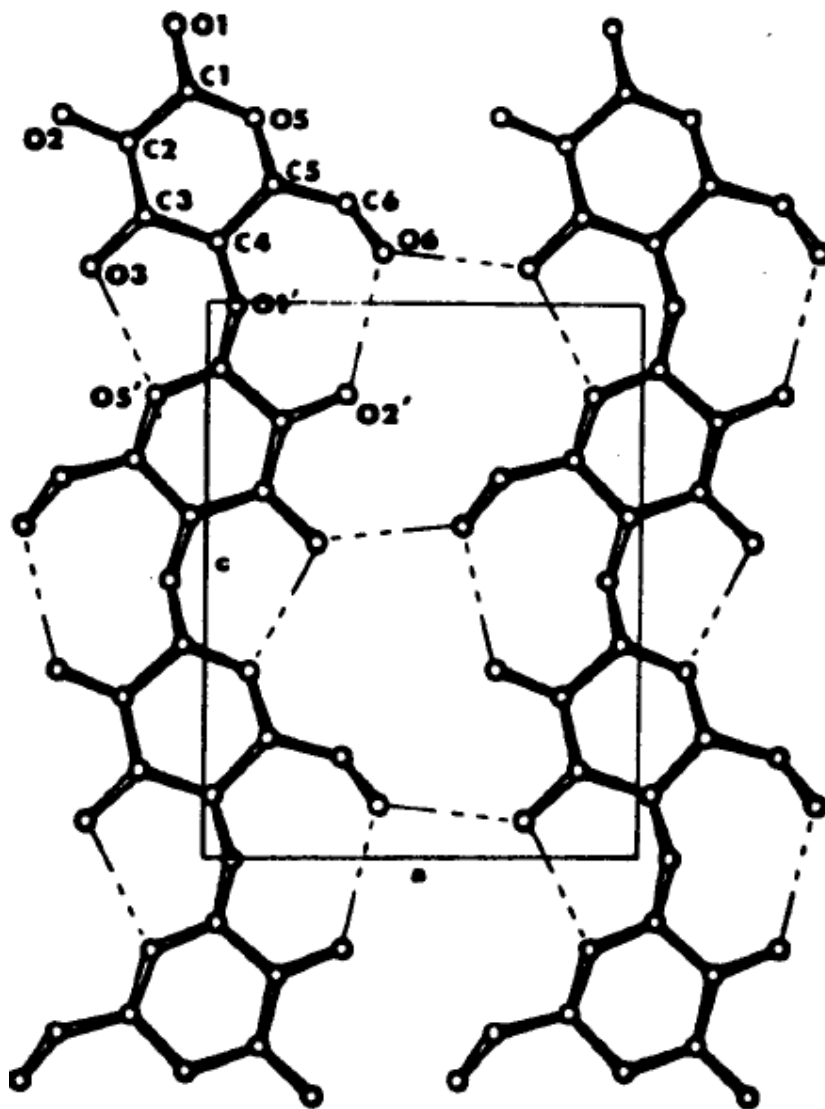
The majority of wood carbohydrates is cellulose, it is a polysaccharide consisting of glucose units. Cellulose is a glucan polymer consisting of linear chains of 1,4-b- bonded anhydroglucose units. The number of sugar units in one molecular chain is referred to as the degree of polymerization (DP), different molecular chains have the different DP values (ROGER C. PETTERSEN. Madison, WI 53705.page 2).

Mercerization is Treatment of alkali cellulose. Regeneration is the treatment with alkali and carbon disulfide, the formation of a soluble fiber Xanthate derivative. The derivative is converted back to cellulose and reprecipitated as regenerated cellulose. The structure of regenerated has space group symmetry P2₁ with $a = 8.01$, $b = 9.04$, $c = 10.36 \text{ \AA}$, and $\gamma = 117.1^\circ$, and two cellobiose moieties per unit cell. The structure of it shows in the picture 2 and 3 in the regenerated the packing arrangement is modified, and there is a more intricate hydrogen-bonded network can extends between layers and within layers. As a result, there is a more thermodynamically stable substance. (American Chemical Society.1984 page 3).

Cellulose is an insoluble substance in most solvents including strong alkali, it is hard to separate cellulose from the wood in pure form, because cellulose is close integrate with lignin and hemicelluloses.(Forest Products Laboratory,)



Picture 2 Structure of Cellulose (American Chemical Society.1984.page 5)



Picture 3 Structure of Cellulose (American Chemical Society.1984.page 5)

2.3.2 Hemicelluloses

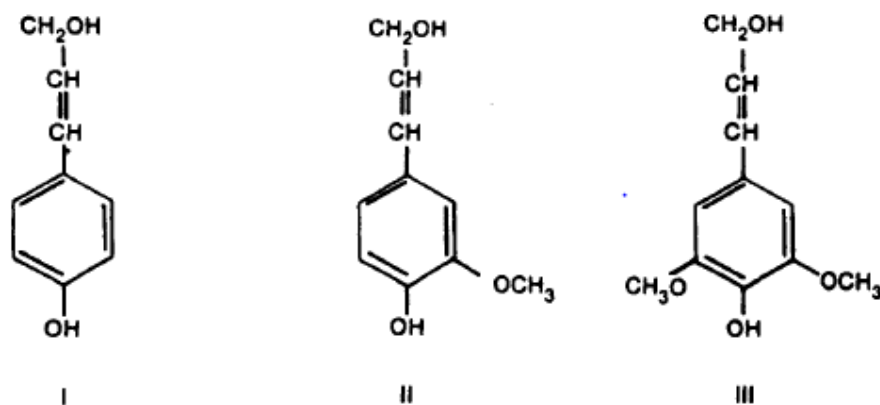
Hemicelluloses are mixtures of polysaccharides synthesized in wood almost entirely from glucose, mannose, galactose, xylose, arabinose, 4-o methylglucuronic acid and galacturonic acid residues. Hemicelluloses are soluble substances in alkali and easily hydrolyzed by acids. Some amounts of rhamnose contain in the hardwood. Normally, cellulose has higher molecular weight than hemicelluloses, hemicelluloses are intimately associated with

cellulose and contribute as a structural component in the plant. There are a lot of hemicelluloses are present when the plant is under pressure. For instance, in the higher pressure the wood have higher galactose and lignin content than normal situation (American Chemical Society.1984.page 6).

The structure of hemicelluloses can be formed with monomer units, there are three entries under each monomer, and in each entry, there are two optical isomers of glyceraldehydes, this is the simplest carbohydrate (American Chemical Society.1984.page 7).

2.3.3 Lignin

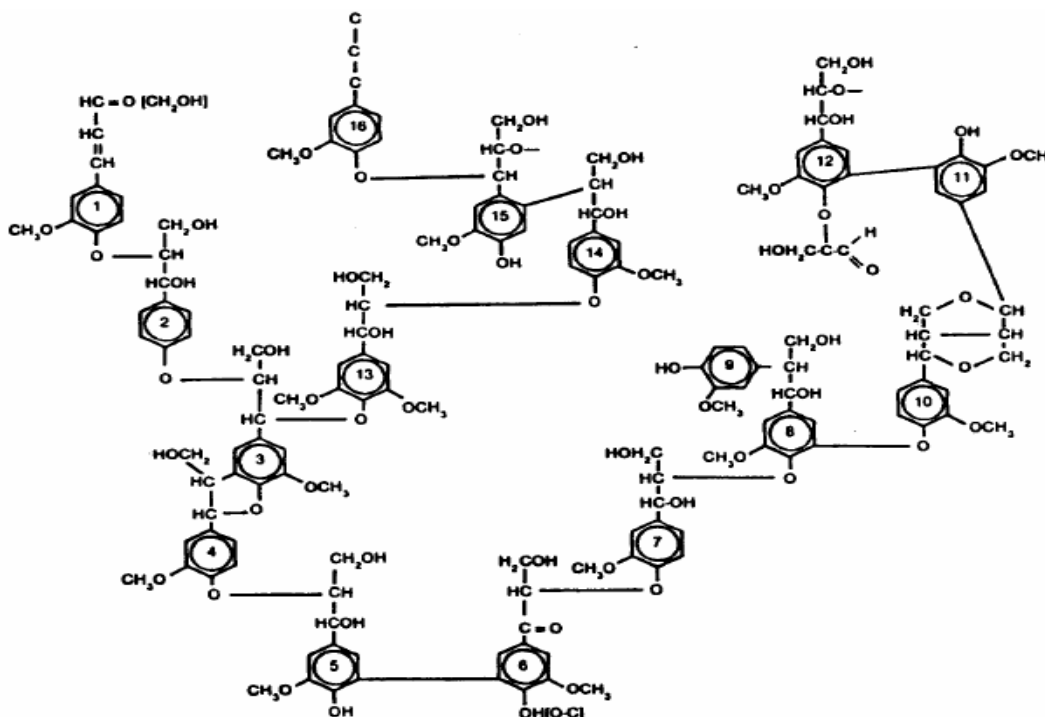
Lignin is a phenolic substance which contains an irregular array of variously bonded hydroxyl and methoxy substituted phenylpropane units. The precursors of lignin biosynthesis are p-coumaryl alcohol (I), coniferyl alcohol (II), and sinapyl alcohol (III). (American Chemical Society.1984.page10) There is a formula showing the structure as follow: picture 2



Picture 4 composition of lignin (American Chemical Society.1984.page 10)

I is a minor precursor of softwood and hardwood lignin; II is the predominant precursor of softwood lignin; and II and III are both precursors of hardwood

lignin. These alcohols are linked in lignin by ether and carbon–carbon bonds. It is a schematic structure of a softwood lignin meant to illustrate the variety of structural components. The 3, 5-dimethoxy-substituted aromatic ring originates from sinapyl alcohol III, and is present only in trace amounts (<1%). Carbon–carbon bonds do not show a lignin-carbohydrate covalent bond. There has been much controversy concerning the existence of this bond, but evidence has been accumulating in its support. (American Chemical Society.1984.page 6) The structure of lignin shows as picture 5:



Picture 5: The structure of lignin (American Chemical Society.1984.page 7)

Lignin can be separated in various methods, in the paper making process there is one step called oxygen delignification. Acid hydrolysis of wood isolates Klason lignin, which can be quantified; however, it is too severely degraded for use in structural studies. (ROGER C. PETTERSEN. Madison, WI 53705.page 7).

2.3.4 Extraneous Components

Extraneous components do not have any contribution for the cell wall structure; most of them can be dissolved in the neutral solvents. Extractives are the extraneous material soluble in neutral solvents, constitute 4-10% of dry weight of normal wood species that grow in temperate climates. At the same time extractives are a variety of organic compounds which contain fats, sugars, resins, starches, glycosides and various proteins. Many of these functions are as intermediates in tree metabolism, as energy reserves, or as part of the tree's defense mechanism against microbial attack. The functions of them are support color, odor, and decay resistance of wood properties. (Forest Products Laboratory)

3 COMPARISONS OF GENERAL PROPERTIES OF BIRCH, PINE AND NORWAY SPRUCE

3.1 General

Wood fiber is a basic raw material in paper production. Fibers are bonded to one another in the wood, wood fibers which are untreated are too stiff and have weak bonds, the purpose of pulp production is to separate fibers from each other, as we know, there are two main methods for producing pulp, they are: chemical pulping, chemicals and heat are used to dissolve or separate fiber binding agent. Mechanical pulping, in which lignin is softened by mechanical stress.

Pine, birch and spruce are the traditionally papermaking raw material, different pulps are produced from different wood types, pulp made from pine species is usually produced using kraft processes, however, spruce is usually used for the mechanical pulps as a material. At the same time birch, pine and spruce as traditional raw materials will find new applications; good bonding capacity, bulk and opacity are required from birch pulp. Specially good strength properties, mainly tensile and tearing strength, all of these aspect are required from softwood pulp. Reinforcement pulp typically includes 70-80 % spruce and 20-30 % pine. A good reinforcement fiber is flexible, long and strong. For example, a lot of reinforcement pulp (40-60 %) is used in LWC paper, but in SC paper it is only used in small amounts (0-20 %). (B.R. Tunstall and B.W.R. Torssell, 2001)

3.2 Fiber Dimensions

Fiber dimensions have a greater impact on the differences between softwood and hardwood pulps than chemical composition. The strength properties of pulp and paper are affected by fiber length. The flexibility of fibers and

tendency to collapse fibers are depending on the fiber width and fiber wall thickness. Fiber size can affect the number of fibers per unit of weight.(B.R. Tunstall and B.W.R. Torssell , 2001)

Fiber dimensions vary within the stem in both vertical and horizontal directions for the same wood; in the mechanical pulp the different grinding angle produce the different fiber dimensions and length. The average length of Finnish birch wood fibers are about 1.1-1.2 mm; the width is about 3 to 4µm. Fiber diameter is appr. 22 microns. Fiber has relatively thin walls which mean it collapses easily. (Sixta, H. 2006 page 44) there is a table shows the dimension of fibers as follow:

Table 1 Dimensions of hardwood and softwood fibers (know pulp)

	Birch	Aspen	Euca-lyptus	Acacia	Pine earlywood/ latewood	Spruce earlywood/ latewood
Fiber lenght, mm	1.1	0.8	1.0	0.7	2.9/2.9	2.9/2.9
Fiber width, µm	22	18	16	20	35/20	33/19
Fiber wall thickness, µm	3	3	3	2	2.1/5.5	2.3/4.5

The pine pulps can be used interchangeably in most printing and writing grades, it is useful for the lighter weight papers. Southern pine pulps have thick fiber walls, due to high latewood content. The southern pine pulps some features which are high tear strength, bulk and absorbency, however, it need much more refining to have a useful tensile strength, compared to Scandinavian softwoods krafts. In the opacity aspect, southern pine pulps have less than other softwood and the formation is more difficulties because of their high coarseness.(B.R. Tunstall and B.W.R. Torssell, 2001)

About 95% of spruce wood materials consists of tracheids which are lignified and death vessel elements, the function of tracheids is to transport water and support the stem. The older spruce tree contains higher coarseness and longer fibers. The function of coarseness has high correlation with the cell wall thickness. The age is apparently not that important for spruce than for pine. Spruce fiber is an excellent raw material for reinforcement pulp with a wide range of age and size classes. Spruce fiber is long and "thin", the length of pine fiber varies, but fiber is "thicker". It can affect all the properties of the spruce fiber, for example: spruce fiber is easily refined, has higher tear strength, tensile strength develop fast and so on. Pine fibers have the opposite properties compared with spruce.(B.R. Tunstall and B.W.R. Torssell,2001)

3.3 Effect of Fiber Dimensions on Paper Technical Properties

The paper technical properties can be affected by the fiber dimensions. A stronger pulp can be produced with long fiber softwood pulp than a short fiber deciduous pulp. However, a certain average length (2-3mm) paper strength is decrease, the strength is determined by the strength of individual fibers. On the other hand, if the fibers are too long it will not produce an even sheet structure and the strength of paper may be compromised. If the fibers are too short it may produce an even sheet structure, but their strength properties will no longer be useful and runnability will be compromised. (C. Ververis , , a, K. Georghiou, 2003)

As we know, the size of fiber is smaller and lower density hardwood fibers than softwood; therefore, the numbers of fibers in hardwood pulp are 6-7 times as there in the same amount of softwood pulp. as a result, the hardwood pulp have some specific properties: better formation, more even sheet surface, smaller pore size and superior opacity. (Ververis , Georghiou, 2003)

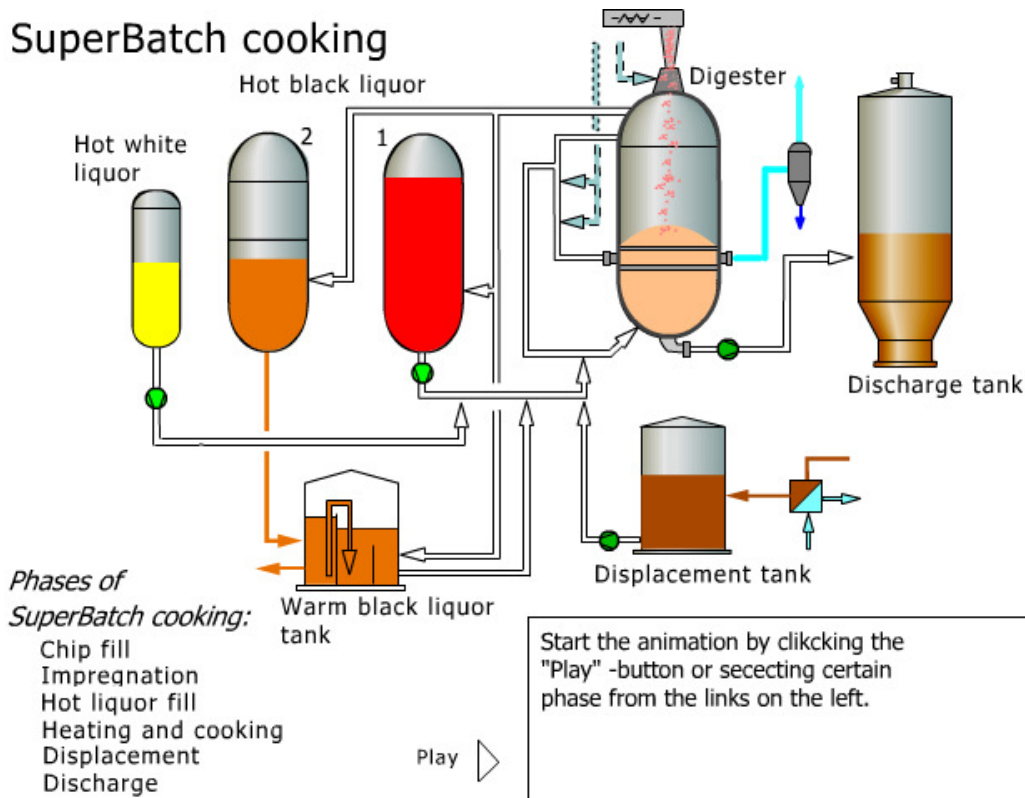
Different wood materials also have big differences in fiber dimension in

hardwood. The birch fibers are bigger than the eucalyptus, their number per unit of weight is higher, so the eucalyptus pulps have better formation and a higher degree of opacity than papers made with birch pulps. The paper made of short fibers with relatively narrow length distribution (eucalyptus, acacia) has also narrowed pore size distribution (small porous) which is favorable for ink absorption. (Ververis , Georghiou, 2003)

4 SULPHATE COOKING AND OXYGEN DELIGNIFICATION

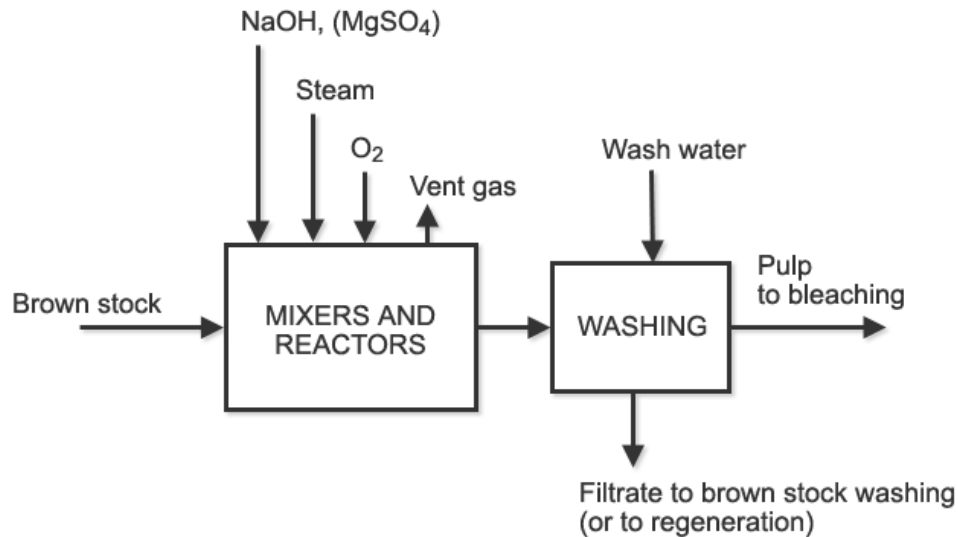
4.1 Introduction of Sulphate Cooking and Oxygen Delignification

The main active chemical agents in the Kraft process are hydroxide (OH^-) and hydrosulfide anions (HS^-) which are present in Kraft cooking liquor, as aqueous solution of caustic sodium hydroxide and sodium sulfide, denoted as white liquor. Wood chips are cooked with caustic soda to produce brown stock, which is then washed with water to remove sulphate cooking (black) liquor for the recovery of chemicals and energy. The batch sulphate cooking plant is used widely in the sulphate cooking process, (Sixta, 2006, p 229-330) generally, the principle of sulphate cooking process is shown as follow picture 6:



Picture 6: Super Batch Cooking (Know Pulp)

Oxygen delignification is a process where part of the residual lignin left in pulp after sulphate cooking is removed using oxygen and alkali. Oxygen delignification is a direct extension of the lignin removal process which takes place during sulphate cooking. The pulp cannot be cooked to a very low kappa number without incurring yield loss in the sulphate cooking process, in the oxygen delignification fragments and oxidizes lignin into a form which is dissolvable in alkali, destroys the color compounds in the lignin and removes impurities (resin) from the pulp. (sixta,2006, pp 259-380).The oxygen delignification stage shows as follow: picture 7



Picture 7: Flow Sheet of Oxygen Delignification (Know Pulp)

4.2 Sulphate Cooking Process

In the batch sulphate cooking pulp is produced in several different digesters, Each digester in turn undergoes chip filling, liquor filling, a heating phase, sulphate cooking and blow. Normally, there are at least four digester are to ensure adequate, different production. (Sixta, 2006, pp 392-482).As we discussed in the introduction part, the batch sulphate cooking stages are as follow:

- Chip filling
- Pre-steaming of chips, equivalent of steaming in continuous sulphate cooking
- White liquor filling, also black liquor added in amounts required by liquor-to-wood ratio
- Heating
- Sulphate cooking, degassing at the same time
- Degassing and blow

The production control is very important for the sulphate cooking process. The

main control problem in displacement batch sulphate cooking, as in conventional batch sulphate cooking, is to sequence the digester operation in such a way that high capacity is achieved (the digester should not have to be kept waiting at a stage) and that overlong cooks, detrimental to pulp quality and uniform quality, are avoided. Quality control is another important point for the sulphate cooking, we need to know the dry wood contain in the digester in order to determine the exact alkali charge. (Sixta, 2006, pp 475-482)

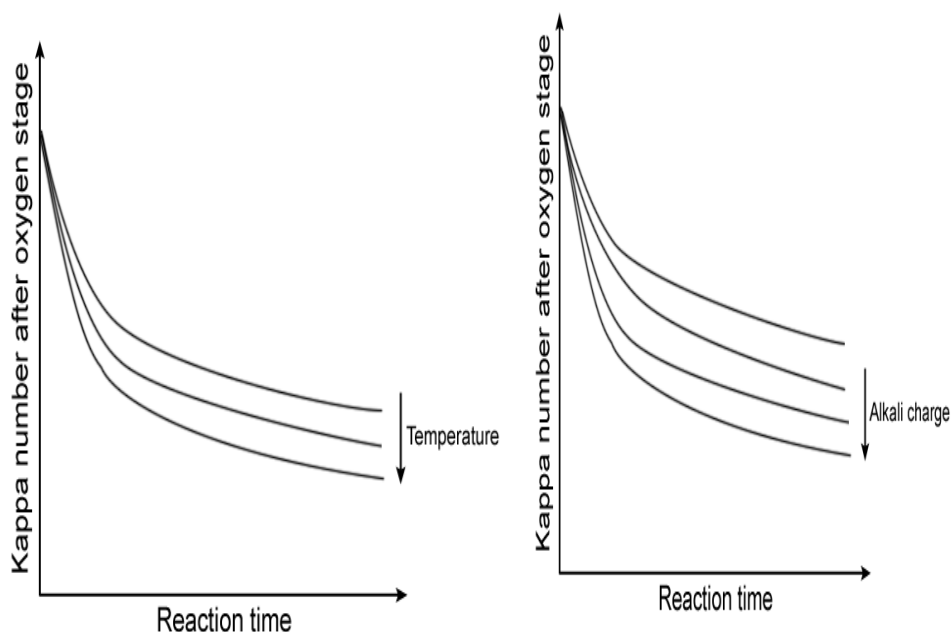
The H-factor calculation is necessary part in batch sulphate cooking, H-factor determine on the sulphate cooking time and temperature. Batch sulphate cooking is stopped generally when a certain H-factor is achieved. H-factor targets can be adjusted on the basis of kappa number measurements. Kappa variations inside the digester can cause difficulties with laboratory and on-line measurements and sampling should be standardized if it is taken from the emptying line of the digester. The differences are evened out in the pulp tank, but if measurements are taken after the pulp tank any possible differences between the digesters will not be made apparent. (sixta, 2006, p 427)

4.3 Oxygen Delignification

Oxygen reactions in an alkaline medium are complex radical chain reactions. Oxygen reacts with both lignin and carbohydrates. Oxygen is reduced to water in reactions with the organic components, and the organic components are oxidized. In the reduction sequence of oxygen can be dissolved by Transition metals. The organic substance dissolved in sulphate cooking creates side reactions in the oxygen stage. This will cause additional oxygen consumption and degradation of cellulose chains. An increase of washing loss before the oxygen stage causes the reduction of pulp viscosity, strength and yield. Correspondingly, efficient post-oxygen washing is the key for low cost bleaching. (Sixta, 2006, p 730)

In the oxygen delignification process the oxygen molecule is able to react only

with certain lignin structures, however, the ionized lignin structure breaks up further and dissolve as the result of chain reaction under the strong alkaline conditions. Oxygen does not react with hexenuronic acids. (sixta, 2006, p 780) Generally speaking, the kappa number of the pulp entering the oxygen delignification is 25-30 for the softwood, but it is less than 20 for the birch. If sulphate cooking is extended to a very low kappa number, the pulp yield from wood as a whole is lower and the pulp strength worse than when delignifying with oxygen to a corresponding kappa number. With the pressure increase to 4 bars oxygen becomes reactive at 75-80 °C. When the temperature is raised, reactions speed up considerably. The same degree of delignification is achieved at a higher temperature with a smaller alkali charge than at a lower temperature. (sixta, 2006, p 260) There is a picture show the effect of oxygen delignification in picture 8



Picture 8 Relationship between kappa number and oxygen delignification (Know Pulp)

5 BEATING PROCESS

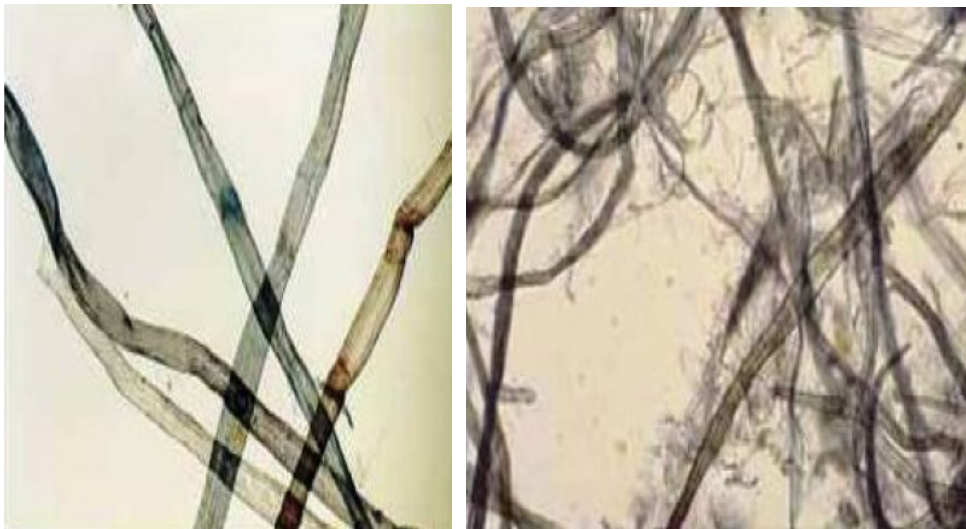
There are several ways to increase the sheet strength and enhance the binding power of plant fibers in practical papermaking process. However, nowadays hammers are used instead of beating, the aqueous fiber slurry is milled by a device called a “valley beater” or “refiner”. After the mechanical treatment, the fiber wall is squeezed, kneaded and plasticized, therefore the fiber surface is partly disintegrated. Mechanical shear separates parts of cellulose fibrils so that the fiber surface becomes hairy and fluffy. Fibrils may involuntarily be completely liberated from the fiber materials and some fibers may even be completely torn to fiber fragments. (Sixta 2006, p 1281)

When testing the properties of paper pulps, there are two different points between sulfite and kraft pulps. The first point is that kraft pulps have a much higher resistance towards beating than do sulfite pulps. The kraft pulps take more time to reach a given drainage resistance than sulfite pulps. Another point is that sheets from sulfite pulps are considerably lower in tear (22%) and burst (17%) strengths, and somewhat lower in tensile (10%) as compared to kraft pulp sheets. Kraft pulps exceed sulfite pulp in tearing strength by 44-80% in the same tensile index. The fiber strength is proportional to the cellulose content, but above a value of 80% cellulose content the fiber strength diminishes due to replacement of flexible cellulose-hemicelluloses-cellulose bond by the more rigid cellulose-cellulose bonds. (Sixta 2006, p1011)

The relationship to the degree of order established by measuring the level-off DP (LODP). Which is a sample means of determining the mean crystalline length. Paracrystalline regions in native cellulose fibrils are transformed into amorphous regions during pulp processing that are viscoelastic and capable of absorbing more energy under mechanical stress. The excellent swelling properties cause by LODP and high modulus conditions. The high beating rate of sulfite pulps were attributed to the greater amounts of crystalline and

paracrystalline regions compared to kraft pulps. The rate at which a pulp beats is virtually a measure of a rate at which the wet cell wall splits and breaks up while applying mechanical stress. Fiber of amorphous structure thus containing viscoelastic properties is more likely to withstand stress without fracture, since the stress will be relaxed and energy dissipated in the viscoelastic regions.(Sixta 2006, p1012)

As we know, the beating can change some single fiber properties. The structure and bonding of paper depends on fiber properties such as fibrillation and also the density and tensile strength of paper therefore increase due to beating. There are indications that fibre/fibre joint strength also increases with beating due to changes in the physical structure of the fiber surface that makes new surfaces available of molecular bonding. There is a picture shows the change of fiber's structure:



Picture 9: Fiber before beating picture 10 Fibers after beating (knowpulp)

Beating dried, hornified pulps to a certain degree of freeness requires significantly higher energy consumption than beating virgin fibers. Beating can make the fibers become brittle and break in the hornification. When comparing dried and non-dried chemical pulps beaten to similar freeness levels, the

non-dried pulps paper sheets have better mechanical properties, however, it has lower light scattering and opacity. For the chemical pulps with a high yield or high lignin content the effect of beating and wet pressing are not the same. Generally speaking, beating and wet pressing still increase density, at a high level of wet pressing, the increase in fines content with beating has no effect on the light scattering. Beating is an efficient way to affect the counteractive bonding ability and fiber flexibility. Beating increases both of these and therefore the elastic modulus of paper increase strongly, but flexible fibers also decrease paper thickness, bending stiffness may not change at all. (Niskanen 2008, p 161)

Laboratory use the PFI mill to predict the usability of a pulp, it is the similar process as the industry beating. Since beating is used in the laboratory, beating in the chemical pulp testing is possibly the most important single phase. The quality of water used has a significant effect on the pulp behaviour and therefore on the beating results. The electrolyte content and temperature, and PH, these factors are very important for the beating results.(Lindstrom&Kolman 1982) An laboratory PFI mill is shown in the picture 9:



Picture 11: PFI mill

6 EXPERIMENTAL PARTS

6.1 Experimental Design and Method

The Norway spruce kraft pulps were pulped in laboratory scale using the circulation batch process. Two different pulps were made, one pulp was after sulphate cooking process, another one was after oxygen delignification treatment. In the pulps analysis process, the kappa number of after sulphate cooking process was 29.15, and the kappa number of oxygen delignification treatment was 16.6. These two different pulps were beaten in five different revolution degrees in PFI mill, these degrees were: 0, 2000, 4000, 6000, 8000. After this step, the pulps were divided into two main different parts which include 10 different pulps. The parameters of experimental setup are shown in the following table:

Table 2 parameter of experimental design

Pulps	Feature of pulp	Kappa number
After sulphate cooking process	Revolution degree: 0, 2000,4000,6000,8000	29.15
After oxygen delignification	Revolution degree: 0, 2000,4000,6000,8000	16.6

As we can see from the table above, these were the main idea of the pulps, however, there were also some other important parameters which can affect the sulphate cooking process, for instance, H-factor, the amount of active alkali(129.44g/L),the temperature and duration of sulphate cooking process were very important.

6.2 Raw Material

Raw materials were came from the Saimaa University of Applied Sciences laboratory. The moisture content of chips was the same for these two pulps, the dry content of chips was about 93%. To prevent the moisture content of chips, they were kept in the refrigerator at temperature of 5°C. After this step, chips were screened in gyratory screen. The purpose of gyratory screen was to obtain the chips with length of 19mm-25mm, and no barks, knots. Some other not useful materials were removed during the chips screening. It can improve the quality of pulps, affect the properties of paper.

6.3 Sulphate Cooking Method and Conditions

The sulphate cooking process was done in the batch digester of tank size 0.01m^3 (10 litres) in the liquid circulated system. The weighed chips(common 1.0-1.5kg calculated as oven dry) were put into the basket, in this experimental the weight of chips was 1075g, sulphate cooking liquor was poured carefully into the sulphate cooking vessel on the chips. According to the relationship between chips and white liquor, the required amount of water was added to maintain wood to liquid ratio at 1:4. The amount of white liquor was 1.92l, the active alkali was 129.44g/l, and alkali charge was 25%. After that, the important point was that cold cooling water was opened for the circulation pump and piping of liquor circulation. All valves were in right positions-pressure in the liquor circulation system cannot risen up after just staring. If the pressure went up too fast, the pumps were stopped and the system was checked so that there were no closed valves, as they must be opened during sulphate cooking. Heat exchanging of sulphate cooking system was turned on, the temperature would fast rise up to 80°C, and it took 75

minutes to raise the temperature of sulphate cooking liquor from 80°C to 170°C, the temperature increased about 1.5°C/min. Homogeneously cooked at 170°C for 88 minutes. The pulp got kappa number 29.15. When the sulphate cooking time finished, heating was turned off, the circulation pump was on. the heat exchanger was cooled instead of heating by opening cooling water valve, the deckle of the digester could not be opened before the pressure was zero. After the pulp was taken out from batch digester tank, washing was necessary. For the washing of pulp, 2% of sodium hydroxide (NaOH) was used; the washing was repeated several times till the liquid was clean.

6.4 Oxygen Delignification and Conditions

In the oxygen delignification process the oxygen molecule was able to react only with certain lignin structures, however, the ionized lignin structure breaks up further and dissolve as the result of chain reaction under the strong alkaline conditions. 200g (bound dry mass) was weighted of spruce pulp, 3% of sodium hydroxide (NaOH) 200ml and 0.5% of magnesium sulfate (MgSO_4) 100mL were impregnated, the consistency was 10%, the total volume was 2l. Preheating pulp and water in microwave or other heat source was necessary. Correct reaction temperature and pressure was setting to be sure that the connection amount of equipment was well. The heating process heated for about 50 minutes. When the oxygen delignification treatment was finished, the pulp was taken out of the tank, after that washing equipment was needed.

6.5 Disintegration and Screening

It was necessary to use the mechanical treatment to separate pulp which was like loosely packed fiber bundles. The laboratory disintegrator was used to separate bundles pulp. After the disintegration treatment, the pulp was screened in Somerville screen with the slots of diameter 0.2mm, the reject pulps were thrown away, the accept pulps were collected in the wire bags. To remove the water, the bag was centrifuged. After that the accept pulp was taken out from the wire bag, transferred to the air tight container and kept in the refrigerator at 5°C.

6.6 Kappa Number Analysis

The Kappa number was an indication of the lignin content or bleaching ability of pulp. It should be noted that there was no general and unambiguous relationship between the kappa number and lignin content of a pulp. The method was applicable to all kinds of unbleached pulp obtained in yields under about 60%. The degree of delignification of pulps produced at higher yields should be determined by methods which do not involve permanganate oxidation, such as ISO 3260. The number of milliliters of 0.02 mol/l potassium permanganate solution consumed under the specified conditions by 1 g of pulp. The results were corrected to a value corresponding to that obtained when 50% of the permanganate was consumed in the test.

2 g dry content pulp was disintegrated the test specimen in not more than 500ml of distilled water until free from fiber clots and from large fiber boundless. The disintegrated test specimen was transferred to a 1500ml reaction beaker and rinse out the disintegrator with sufficient water to bring the total volume to 790ml. with a pipette, 100ml of potassium permanganate solution and 100ml of

the sulphuric acid were added to a 250ml beaker. This mixture was brought to 25°C and added it quickly to the disintegrated test specimen and simultaneously the timing watch was started. The 250ml beaker was rinsed, at least 10ml of distilled water was used, and the washing was added to the reaction mixture. The total volume was 1000ml.at the end of exactly 10 minutes; the reaction was terminated by adding 20 ml of the potassium iodide solution from a graduate cylinder. Immediately after mixing, but without filtering out the fibers,the free iodine with the sodium thiosulphate was titrated cooking solution. A few drops of starch indicator solution was added toward the end of titration. A blank test was carried out; the exactly same procedure was used as above.

The kappa number X expressed as a numerical value only, was given by the formulae:

$$V_1 = \frac{(V_2 - V_3)c}{0.02 \times 5} \quad (\text{ISO 302-1981})$$

$$X = \frac{V_1 d}{m}$$

Where

V_1 was the volume of the potassium permanganate solution consumed

V_2 was the volume of the sodium thiosulphate cooking solution consumed in blank test

V_3 was the volume of the sodium thiosulphate cooking solution consumed in determination in millilitres

c was the concentration of sodium thiosulphate cooking solution in moles per litres

d was the correction factor to 50% permanganate consumption,

m was the oven dry mass of the test specimen, in grams

There is a table showing the correction factor d, expressed as a function of V_1

Table 3: Correction factor d, expressed as a function of V_1 (ISO 302-1981)

V_1 ml	d									
	0	1	2	3	4	5	6	7	8	9
30	0.958	0.960	0.962	0.964	0.966	0.968	0.970	0.973	0.975	0.977
40	0.979	0.981	0.983	0.985	0.987	0.989	0.991	0.994	0.996	0.998
50	1.000	1.002	1.004	1.006	1.009	1.011	1.013	1.015	1.017	1.019
60	1.022	1.024	1.026	1.028	1.030	1.033	1.036	1.037	1.039	1.042
70	1.044									

According to the expression of Kappa number formula:

After sulphate cooking pulp kappa number was 29.15, and after oxygen delignification was 16.6

6.7 Beating

The accept pulp was beaten in PFI mill by the beater rotation. A measured amount of pulp of specified stock concentration was beaten between a roll with a bar and a smooth beater housing, both rotating in the same direction, but at a different peripheral speeds. The main part of the beating energy transfers to the pulp via bar surface not via the edges. Compared with a normal low consistency beating of about 2 %-5% in paper mill, in the PFI mill the beating consistency was 10%, being higher than in the paper mill.

In the beating process, about 30 ± 0.5 g of dry pulp at 10% consistency was taken. There were 5 different revolutions for the sulphate cooking pulp and oxygen delignification pulp. The revolution degrees were: 0, 2000, 4000, 6000, and 8000. After beating process 10 different pulp groups were produced which

are shown in table 2(page 26)

6.8 Pulp Properties Test

The pulp properties test which includes the drain ability of a pulp suspension in water (Schopper-Riegler number), water retention value (WRV) and fiber length test were measured for each group. The test results are listed in table 6(page 38)

6.9 Paper Sheet Making

The laboratory paper sheets were made by using “Rapid-Köthen” sheet former. The basic weight of the paper sheets was 60g/m^2 , there were six paper sheets made, which were within $\pm 3\%$ range of 60g/m^2 . The dry content of fibers are about 1.88g per sheet, it means there were 60 paper sheets are about 120 g of dry fibers were needed.

6.10 Paper Sheet Test

Optical properties and strength properties were measured from the sheets. The following strength properties were measured from the sheets.

- Tensile strength
- Tearing strength
- Thickness
- Bursting strength

And optical properties were

- Brightness
- Opacity

7 RESULTS AND DISCUSSION

In this part, the different pulps were compared in the different degree and beating revolution, the main point was that the regularity of pulps was found between the after sulphate cooking and oxygen delignification.

According to the analysis kappa number we knew that the kappa number of sulphate cooking pulp is 29.15, and the kappa number of oxygen delignification was 16.6. Usually the kappa number of the pulp entering the oxygen delignification stage was 25 –30 for softwood and less than 20 for birch. If sulphate cooking was extended to a very low kappa number, the pulp yield from wood as a whole was lower and the pulp strength worse than when delignifying with oxygen to a corresponding kappa number. Generally speaking, it was normal situation to get smaller yields for the later one as the sulphate cooking time was extended to dissolve as much as possible.

Comparison between Schopper-Riegler measurements

Schopper-Riegler number of pulp was called SR-number, the measurement principle of SR number was the same as CSF. SR-number was measured by filtrating 1 liter of stock through the wire of the apparatus. SR number of pulp was the higher the faster the infiltration slowed down influencing on the fiber cake that developed to the screen. Generally, the functionality of the tester was tested by pouring 1l of the 20°C clean water into the chamber and immediately releasing the sealing plug. The SR-number was read from the scale of the graduated glass when the water has stopped to flow from the side orifice. The scale on which was a discharge of 1000ml corresponds to a SR-number of zero and zero discharge to SR-number of 100. In this pulp test, the

consistency of pulp was 2 g/l, the test was performed following the procedures strictly stated on ISO 5267/1-1979(E), All the test value are shown in the following table :

Table 4: SR-number [SR°] revolutions in PFI mill

Revolution	After sulphate cooking SR°	After oxygen delignification SR°
0	14.0	13.5
2000	15.0	15.0
4000	16.0	16.5
6000	19.0	22.5
8000	23.0	27.0

According to the table 4 was made the chart 1 as follows:

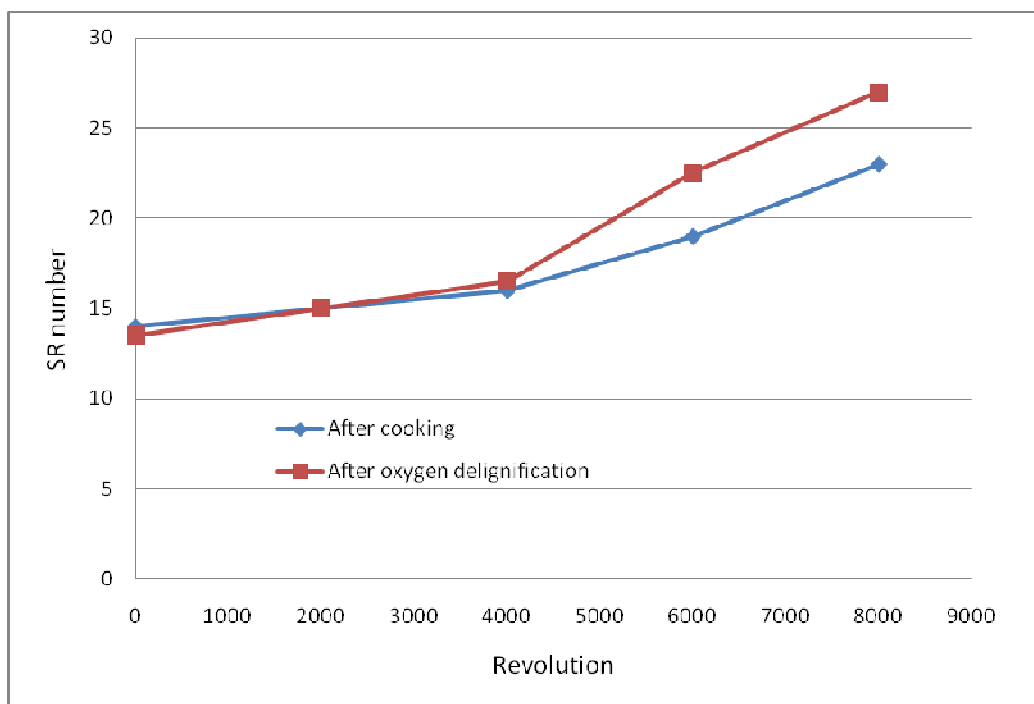


Chart 1 SR-number versus number of revolution in PFI mill

In the chart 1 some rules were found: when the revolution increases, the SR-number increases too. When the revolution is less than 4000, these two pulps had the similar SR-number which was from 14 to 16. However, after oxygen delignification pulp increase sharply in SR-number when the revolution more 4000. When its revolution was 8000, the SR-number reached 27. After sulphate cooking pulp rapidly increased, when its revolution was 8000, the SR-number was 23.

Fiber length

Fiber length was relatively easy to measure in-line. Fiber length and length distribution were the most important quantities when the quality potential of the pulp was assessed in paper industry and was therefore used as a process control variable. The properties of fiber determine 80% of the final paper quality, there were fiber length analyzers based on different techniques. In our laboratory fiber length was measured by “Kajaani FS 300”, it can measure the effect of the different beating revolution on fibers. 0.4 g of dry fibers were taken from each pulp sample, and then it was diluted into 5 l, after that 50 ml of pulp was taken out from the 5 l pulp. After the measuring process, the result of fiber length was shown as follow table 5:

Table 5 Fiber length in Kajaai 300

Revolution	After Sulphate cooking L_c (l) mm	After Oxygen delignification L_c (l) mm
0	3.03	2.78
2000	2.74	2.67
4000	2.64	2.61
6000	2.47	2.52
8000	2.42	2.46

Based on the table 5 was made the chart 2 shown below:

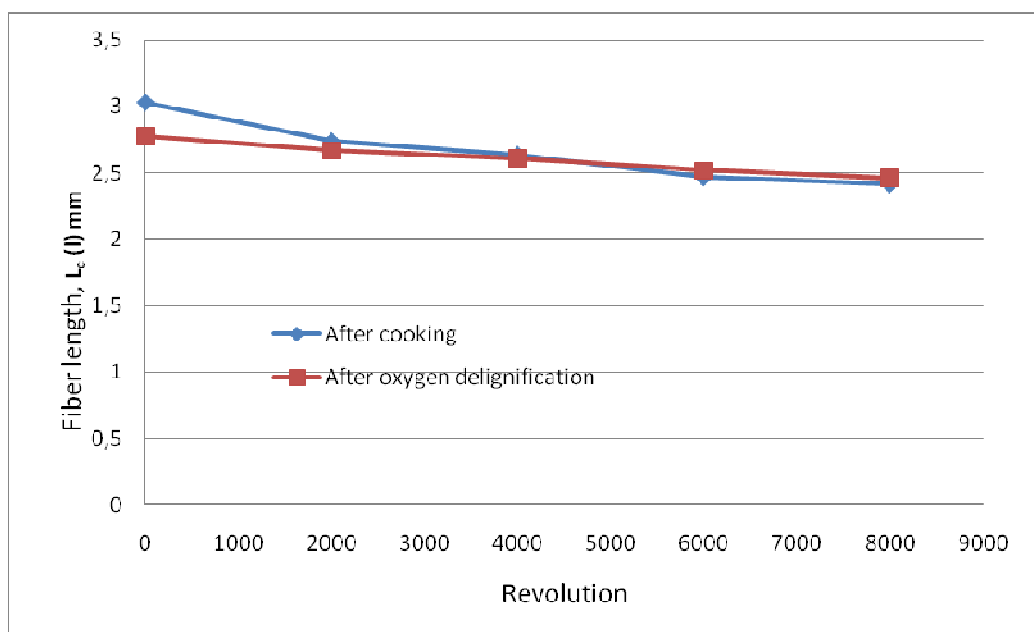


Chart 2 Fiber length versus number of revolutions in PFI mill

Apparent from the graph was that fiber length decreased with the increasing number of revolutions, the reason was fiber breakage while beating. However,

as revolutions were increased the fiber length decreased slowly. The maximum decrease in fiber length was 19% for the after sulphate cooking pulp, for the after oxygen delignification pulp maximum decrease was 9%. It means that the beating process did not have an impressive effect on fiber length. The after sulphate cooking pulp and after oxygen delignification pulp were compared, after sulphate cooking pulp has longer fiber length than the oxygen delignification pulp, the longest fiber length was 3.03mm. For the after oxygen delignification pulp which has the longest fiber length was 2.78.

Water retention

The Water Retention Value (WRV) was an empirical measure of the capacity of test pad of fibers to hold water. The WRV-value increased with increasing beating, the reason was that internal fibrillation is widening of the small internal pores and delaminating, which has been called “swelling” and which occurs concurrently with the development of external fibrils, which also serve to hold additional water. The pad consisting of pulp fibers was formed by dewatering a pulp suspension on a wire screen the test pad was centrifuged under a specified centrifugal force for 10 minutes, weighed, dried and weighed again. The water retention value was calculated from the wet mass of the centrifuged test pad and the dry mass of the test pad. Water retention values were measured in the experiment as shown in the following table:

Table 6 Water retention value (%)

Revolution	After sulphate cooking WRV, %	After oxygen delignification WRV, %
0	160	141
2000	186	164
4000	201	173
6000	214	183
8000	223	195

Based on the table 6 was made the chart 3 as below:

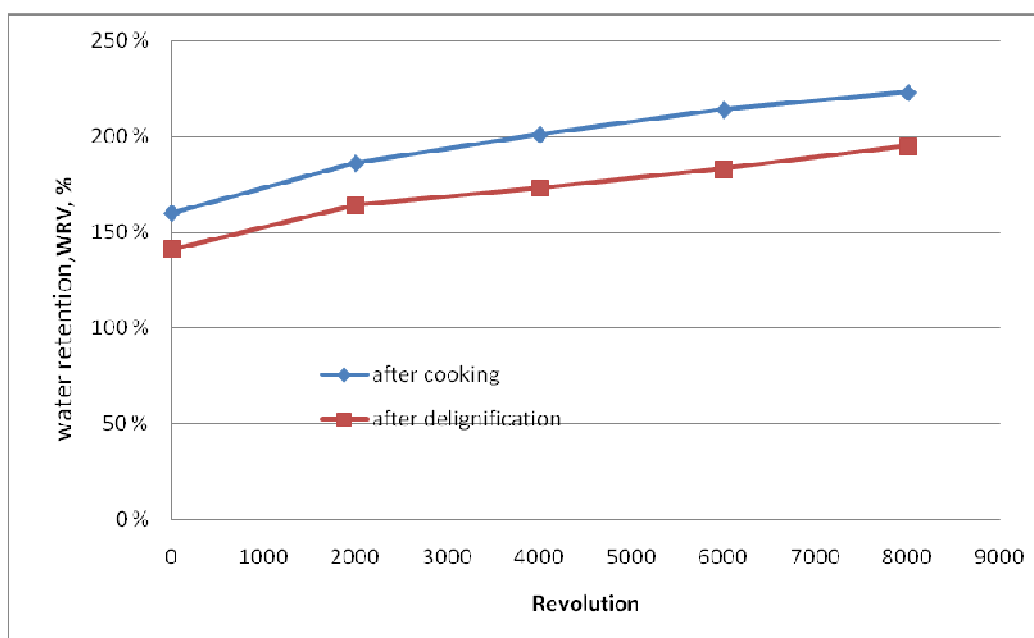


Chart 3 Water retention versus number of revolution in PFI mill

Apparent from the graph was that with the revolution increase, the water retention value increased for these two pulps. It means that the water retention

value can be improved by the beating process. According to the chart 4 that after sulphate cooking pulp had higher water retention than the after oxygen delignification, the water retention value ranged from 140% to 223%. In further beatings, water retention increments were 29 % maximum increase from the unbeaten to the revolution 8000 in sulphate cooking pulp.

Tensile strength

Tensile strength of paper was the highest force that the paper strip can stand without breaking when applying the load in a direction parallel to the length of the strip. Tensile strength can be measured with a horizontal tensile testing machine using constant rate of elongation. The sample of 15mm width and 18-20 cm length was used for the tests. A tensile strength tester measures the load applied as a function of elongation of the sample trip giving a load-elongation. The values of the tensile strength are showing in the following table.

Table 7: tensile strength index

Revolution	After sulphate cooking Tensile index (Nm/g)	After oxygen delignification Tensile index (Nm/g)
0	36.38	36,00
2000	46.55	57.33
4000	51.17	63.44
6000	57.06	68.06
8000	63.13	70.22

Based on the table 7 was made the chart 4 shown below

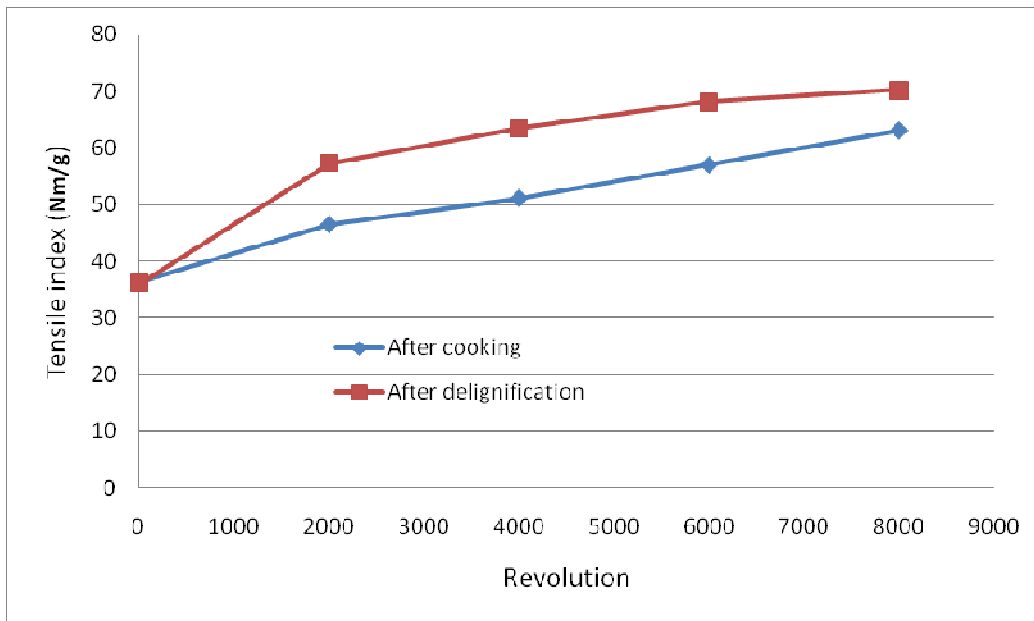


Chart 4: Tensile strength versus number of revolutions in PFI mill

According to the chart 4, the tensile strength increases with the revolution increase, the after sulphate cooking pulp has the lower tensile strength than the after oxygen delignification pulp. It means that the amount of lignin content can decrease the tensile strength. However, when the revolution was 0, these two pulps have almost the same tensile strength; there was a significant increase in paper strength, when comparing the beaten and unbeaten fibers, especially for the after sulphate cooking pulp. The maximum increase in tensile strength was 50% for the after oxygen delignification pulp, the maximum increase in tensile strength was 42% for the after sulphate cooking pulp.

Tear strength

The tearing strength of paper was the mean force required to continue the tearing of paper from an initial cut in a single sheet. Tearing strength simulates the situation when there was some defect in the border of the paper web, then the strength of continues tear was smaller that it was in the whole paper web. In the experiment, tearing strength was measured using “DIGI-TEAR”, a sample of 62mm length and 50 mm width was used for the measurement. The unit of the tear strength was mNm^2/g . The values of the tensile strength are shown in the following table

Table 8: Value of tear index

Revolution	After sulphate cooking tear index (mNm^2/g)	After oxygen delignification tear index (mNm^2/g)
0	7.65	12.46
2000	12.06	14.41
4000	11.90	12.16
6000	13.70	12.46
8000	12.15	11.80

Based on the table 8 was made the chart 5 shown below

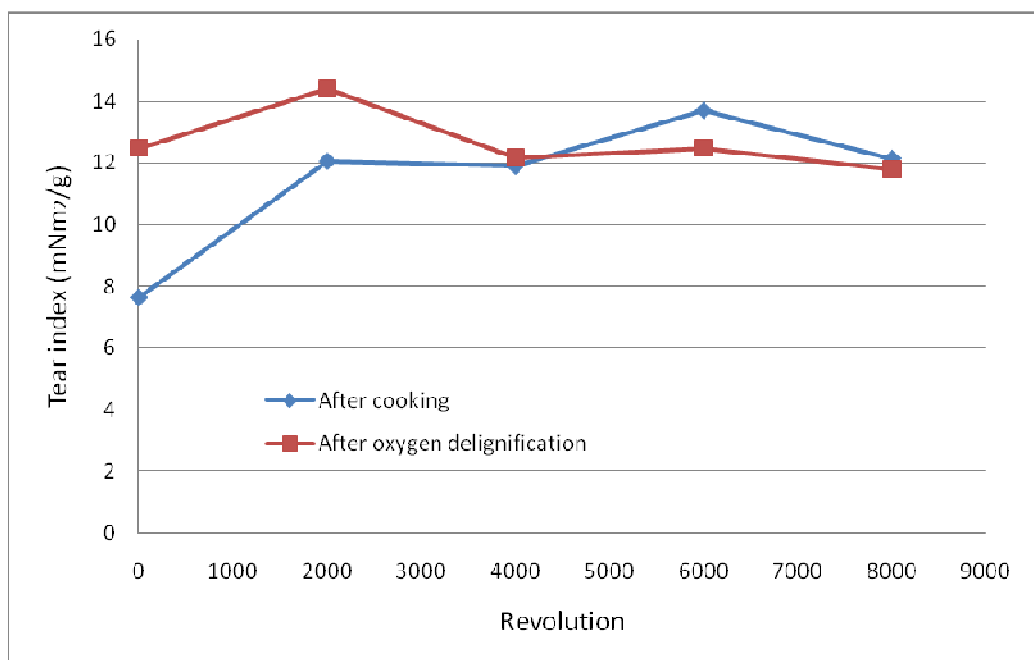


Chart 5: Tear strength versus number of revolution in PFI mill

From the chart above the rules were found that the tear strength of after sulphate cooking pulp paper increases sharply at the first beating by more than 30%, after revolution 2000 the tear strength increases stable, however the after oxygen delignification pulp paper decrease gradually. it means that revolution degree does not have too much effect for the paper's tear strength.

Burst strength

Bursting strength tells how much pressure paper can tolerate before rupture. Bursting strength was measured as the maximum hydrostatic pressure required to rupture the sample by constantly increasing the pressure applied through a rubber diaphragm on 1.20 - inch diameter (30.5 mm) sample. There were five measurements were taken from each sample to give the average value of burst strength. The values of burst strength are shown in the following

table.

Table 9 Strength value of burst strength index

Revolution	After sulphate cooking Burst index (kPam ² /g)	After oxygen delignification burst index(kPam ² /g)
0	2.25	2.21
2000	3.60	3.90
4000	3.90	4.13
6000	3.66	5.35
8000	3.76	5.00

Based on the table 9 was made the chart 6 shown below

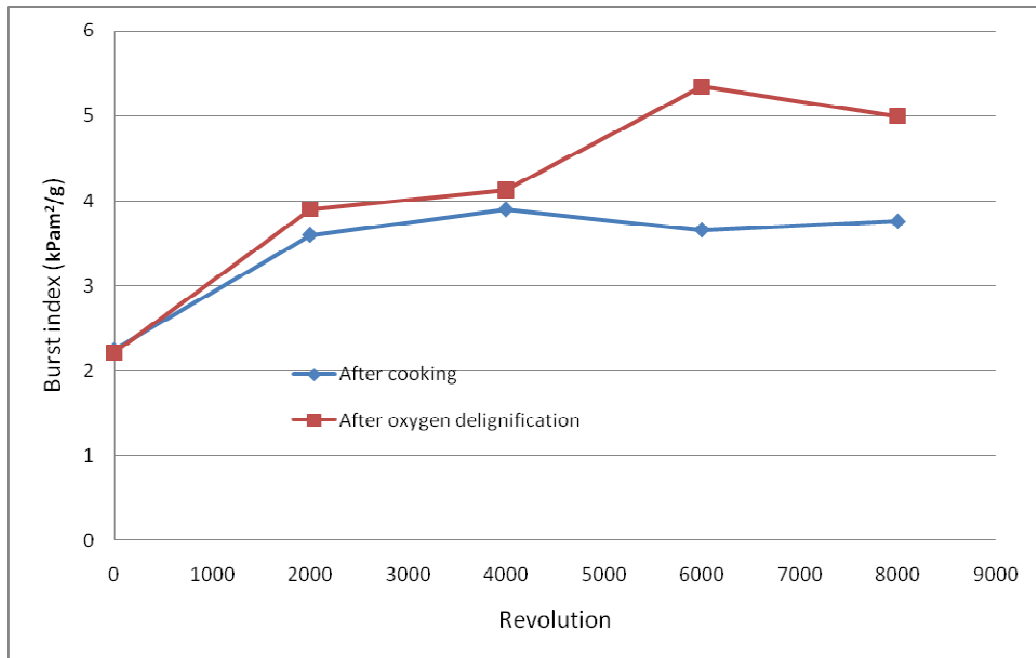


Chart 6: Burst strength versus number of revolution in PFI mill

From chart 6 rules were found that with revolution increase, burst strength increases in both pulps. However, the difference was that after the revolution

2000, the after sulphate cooking pulp paper's burst strength remains stable, meaning that higher revolution beating process does not affect burst strength too much, the after oxygen delignification pulp paper has higher burst strength than sulphate cooking pulp paper, after it reaches the peak, burst strength of after oxygen delignification starts to decrease.

Bulk

Bulk was a very important parameter of paper particularly for printers. Bulk was a term used to indicate volume or thickness in relation to weight. It was the reciprocal of density (weight per unit volume). It was calculated from caliper and basis weight. Decrease in bulk or in other words increase in density makes the sheet smoother, glossier, less opaque, darker, lower in strength etc. In the laboratory the "L&W Micrometer 51" was used to measure 6 different locations on paper sheet. The values of bulk are shown in the following table:

Table 10: Bulk value

Revolution	After sulphate cooking Bulk (cm ³ /g)	After oxygen delignification Bulk (cm ³ /g)
0	1.78	1.62
2000	1.45	1.42
4000	1.39	1.34
6000	1.36	1.28
8000	1.28	1.25

Based on the table 10 was made the chart 7 shown below

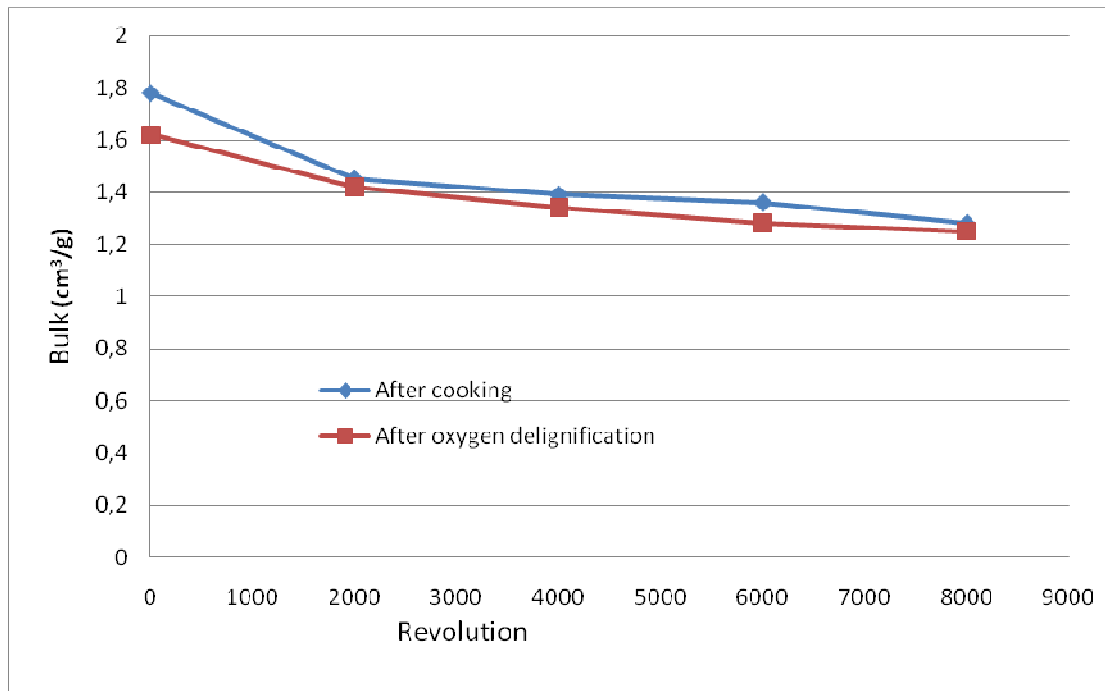


Chart 7: Bulk value versus number of revolutions in PFI mill

Apparent from the graph was that bulk decreases continuously with the increased amount of beating. After sulphate cooking pulp paper decrease 28% through the beating process, another pulp paper decrease 22% after beating treatment. And the after sulphate cooking pulp paper's bulk value is higher than the oxygen delignification pulp paper, but the difference was very small. It means that the sheets made from high lignin containing pulp provided higher bulk value.

Optical properties

Brightness is defined as the percentage reflectance of blue light only at a wavelength of 457 nm. Whiteness refers to the extent that paper diffusely reflects light of all wave lengths throughout the visible spectrum. Whiteness is an appearance term. Brightness is arbitrarily defined, but carefully standardized, blue reflectance that was used throughout the pulp and paper industry for the control of mill processes and in certain types of research and development programs. Brightness is not whiteness. However, the brightness values of the pulps and pigments going into the paper provide an excellent measure of the maximum whiteness that can be achieved with proper tinting (HARI GOYAL, 2202 RUSSET CRS, 2000).” Lorentzen & Wettre – Elrepho” was used to measure optical properties. The values of brightness are shown in the follow table:

Table 11: Brightness value (%)

Revolution	After sulphate cooking Brightness, R457 C	After oxygen delignification Brightness, R457 C
0	26.53	30.18
2000	22.67	26.45
4000	22.07	25.49
6000	21.51	25.24
8000	21.34	25.48

Based on the table 11 was made the chart 8 shown below

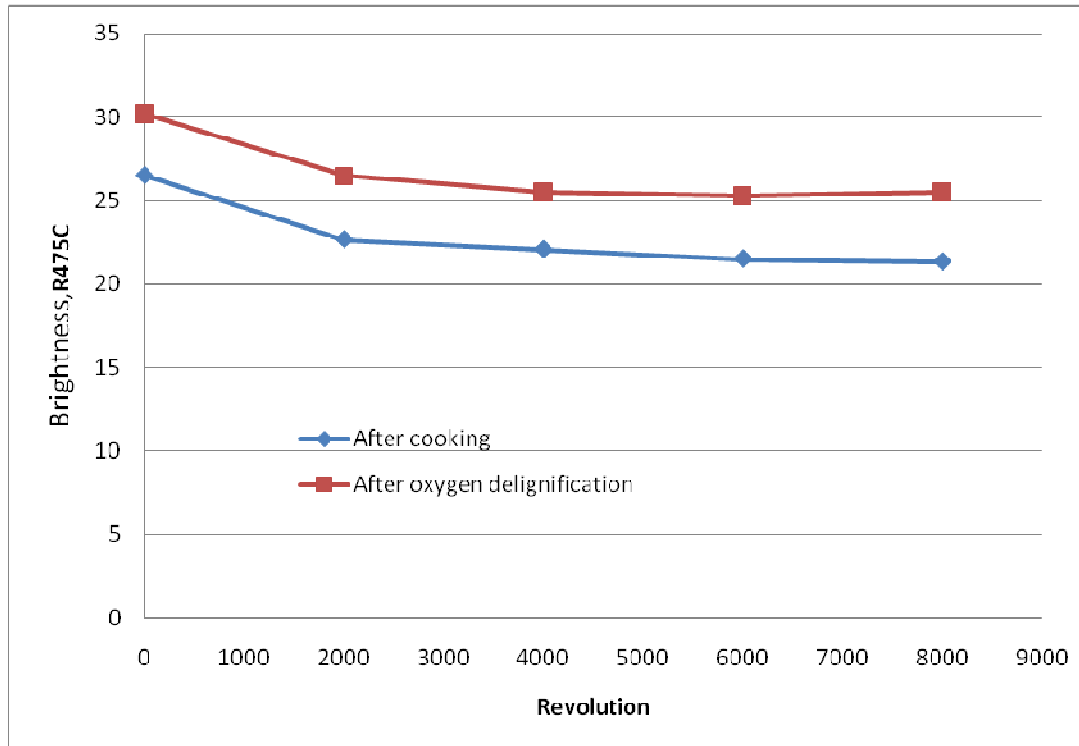


Chart 8: Brightness value versus number of revolution in PFI mill

As can be seen in the chart 8, the brightness value decreases for both pulps as the revolution increase, after sulphate cooking pulp paper's brightness value was lower than after oxygen delignification when pulps have the same beating revolution, but the difference was not so big. As a result, the amount of lignin did not have a big effect on the paper's brightness.

8 SUMMARIES

In this laboratory experiment there were two different pulps which were after sulphate cooking and after oxygen delignification. The chips were cooked in the laboratory scale in a batch digester with sulphate cooking liquor which was white liquor. After that some amount of processed pulps was taken for the oxygen delignification, to remove the lignin from the pulp, this treatment contained 3% of sodium hydroxide (NaOH) 200ml and 0.5% of magnesium sulfate (MgSO_4) 100ml were impregnated, the consistency was 10%, the total volume was 2l. As a result, there were two different Kraft Norway spruce pulps were made. In the washing process, 2% of sodium hydroxide (NaOH) was used; after that the two different pulps were divided into 10 different groups which contained 30 g of dry fibers in each group. These pulps were beaten to 0, 2000, 4000, 6000 and 8000 revolutions by the PFI mill in the laboratory. SR-number, water retention and fiber length were measured. According to the pulp properties test, these pulps were fine which can be used. As a result, the paper sheets were made 60 pieces. However, the paper making process had a little bit different compare with normal way, the different was that after fibers formed paper sheet in the machines, pressing paper sheets were pressed in 4 bars, and then drying process. The paper sheets strength properties and optical properties test was done after papers were fine.

According to the analysis of paper sheets strength properties, some distinct differences were found easily. Unbeaten pulps paper has the lower tensile strength, tear strength and burst strength than the beaten pulps paper, but it has higher bulk value and brightness. Generally speaking, as the beating revolution increase, the paper's tensile strength and burst strength increase gradually, on another hand, its bulk and bright value decrease gradually.

Comparing the difference between after sulphate cooking process and after oxygen delignification treatment with pulps paper sheet test, some rules were found that the after sulphate cooking pulps paper had lower tensile strength, tear strength, burst strength and brightness than the oxygen delignification pulps paper. But the bulk value was opposite. Compare these two different pulps some regular has found that after sulphate cooking pulps had the lower SR-number than after delignification when the pulps at the same beating revolution, at the same time, as the beaten revolution increase, the SR number increase, the fiber length and water retention were higher than oxygen delignification at the same beating revolution level. As the beaten revolution increase, the SR number and water retention increase, however, fiber length decreased.

From this experiment some rules were found out that the paper's properties were expressively affected by beating, beating had a negative effect on bulk and brightness value, it had positive increments in tensile strength, tear strength and burst strength.

Picture 1 The shape of Norway spruce, Page 6

Picture 2 Structure of cellulose, page 5.

Picture 3 Structure of cellulose, page 5.

Picture 4 Composition of lignin ,page 10

Picture 5 The structure of lignin, page 11

Picture 6 Super Batch cooking (know pulp) page 16

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Picture 8 Relationship between kappa number and oxygen delignification ,
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